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<b>(54) Title:</b> HAIR CONDITIONING COMPOSITION  <b>(57) Abstract</b>  A hair conditioning composition comprising: (a) a quaternary ammonium cationic conditioning agent having the formula (I): $[RC(O)OC_2H_4]_nN^+(R^1)_mX^-$ , wherein each R in a compound is a C <sub>6</sub> -C <sub>22</sub> , preferably with only minimal, or no C <sub>6</sub> - <sub>10</sub> , hydrocarbyl group, preferably having an Iodine Value (hereinafter also referred to as IV) of from about 70 to about 140 based upon the IV of the equivalent fatty acid, n is a number from 1 to 3 on the weight average in any mixture of compounds, each R <sup>1</sup> in a compound is a C <sub>1-3</sub> alkyl or hydroxy alkyl group, the total of n and the number of R <sup>1</sup> groups that are hydroxyethyl groups equaling 3, n+m equaling 4, and X is a hair conditioner compatible anion, preferably methyl sulphate, the conditioning agent preferably having (a) a Hunter L transmission of at least about 85, (b) non-detectable levels at the conditions of use of odorous compounds selected from isopropyl acetate, 2,2'-ethylidenebis(oxy)bispropane, and/or short fatty acid esters or (c) preferably both, wherein preferably the level of conditioning agent containing polyunsaturated alkylene groups being at least about 3 % by weight of the total conditioning agent present and wherein preferably the conditioning agent comprises a mixture of monoester and diester; (b) a mono-ol or diol solvent having a ClogP of from about 0.15 to about 0.64; and (c) 1,3-butylene glycol. The compositions herein provide excellent hair detangling, improved wet clean feel on rinsing and post rinsing, and minimize volume loss.		

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## Hair Conditioning Composition

### Technical Field

The present invention relates to a hair conditioning composition.

### Background of the Invention

Scalp hair becomes soiled due to its contact with the surrounding environment and from sebum secreted from the hair follicles. The build-up of sebum and environmental soiling can cause the hair to have a dirty or greasy feel, and an unattractive appearance. In order to ameliorate these effects, it is necessary to shampoo the hair regularly.

Shampooing the hair removes excess sebum and other environmental soiling. However, the shampooing process has disadvantages in that the hair is left in a wet, tangled and generally unmanageable state. Shampooing can also result in the hair becoming dry and frizzy, and a loss of lustre, due to removal of natural oils or other hair moisturizing materials. After shampooing, the hair can also suffer from a loss of "softness" perceived by the user upon drying. The hair can also suffer from increased levels of static upon drying after shampooing. This can interfere with combing and can result in fly-away hair.

A variety of approaches have been developed to condition the hair. These range from post-shampooing hair rinses, to leave-on hair conditioners, to the inclusion of hair conditioning components in shampoos. There are several advantages for providing hair conditioning components such as silicones in shampoos. First of all many consumers prefer the ease and convenience of a shampoo which includes conditioners. An additional advantage of conditioning shampoo compositions is that such compositions provide a good in-use wet hair feel compared to shampoos which do not contain conditioning ingredients. A substantial proportion of consumers however prefer the more conventional conditioner formulations which are applied to the hair as a separate step from shampooing, usually subsequent to shampooing. These hair conditioners typically are formulated as a thickened product, such as a gel or cream, for ease of dispensing and application to the hair.

Consumers who have fine or straight hair may ideally prefer conditioners which provide increased hair volume at the same time as providing conditioning/detangling benefits. Many conventional conditioning compositions based on fatty alcohols and many conditioning shampoos relying upon conditioning ingredients such as silicones, although effective at providing a detangling benefit, cause a decrease in dry hair volume. It would therefore be desirable to provide a hair conditioning composition which minimized decrease in dry hair volume following conditioning.

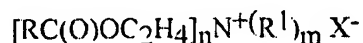
It would be even more desirable to provide a hair conditioning composition which provides good in-use wet hair feel, a wet clean feel on rinsing and post rinsing, dry clean feel, good wet detangling and which minimizes volume loss typically experienced with conditioning conditioners.

It has now surprisingly been found that a conditioning composition comprising a particular cationic conditioning agent, a particular solvent and 1,3-butylene glycol provides a good clean feel on rinsing and post rinsing, good dry clean feel, together with excellent detangling and minimized hair volume loss typically experienced with conventional conditioning compositions.

#### Summary of the Invention

According to the present invention there is provided A hair conditioning composition comprising:

- (a) a quaternary ammonium cationic conditioning agent having the formula (I):



wherein each R in a compound is a C<sub>6</sub>-C<sub>22</sub>, preferably with only minimal, or no, C<sub>6</sub>-10, hydrocarbyl group, preferably having an Iodine Value (hereinafter also referred to as IV) of from about 70 to about 140 based upon the IV of the equivalent fatty acid, n is a number from 1 to three on the weight average in any mixture of compounds, each R<sup>1</sup> in a compound is a C<sub>1-3</sub> alkyl or hydroxy alkyl group, the total of n and the number of R<sup>1</sup> groups that are hydroxyethyl groups equaling 3, n+m equaling 4, and X is a hair conditioner compatible anion, preferably methyl sulfate, the conditioning agent preferably having (a) a Hunter L transmission of at least about 85, (b) non-detectable

levels at the conditions of use of odorous compounds selected from isopropyl acetate, 2,2'-ethylidenebis(oxy)bispropane, and/or short fatty acid esters or (c) preferably both, wherein preferably the level of conditioning agent containing polyunsaturated alkylene groups being at least about 3% by weight of the total conditioning agent present and preferably wherein the conditioning agent comprises a mixture of monoester and diester.

(b) a mono-ol or diol solvent having a ClogP of from about 0.15 to about 0.64;

(c) 1,3-butylene glycol

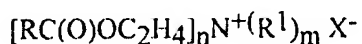
The conditioning composition of the present invention provides excellent in-use wet feel, excellent clean feel during rinsing and post-rinsing, good dry clean feel and excellent detangling, in addition to minimizing volume loss versus conventional conditioning compositions.

The concentrations and ratios herein are by weight of composition, unless otherwise specified. Surfactant chain lengths are also on a weight average chain length basis, unless otherwise specified.

#### Detailed Description of the Invention

The conditioning composition of the present invention comprises a quaternary ammonium conditioning agent, a solvent having a clogP value of from about 0.15 to about 0.64 and 1,3-butylene glycol.

An essential substituted quaternary ammonium compound for use herein may be defined a Diester Quaternary Ammonium active (DEQA) selected from compounds having the formula:



wherein each R in a compound is a C<sub>6</sub>-C<sub>22</sub> hydrocarbyl group, preferably having an IV from about 70 to about 140 based upon the IV of the equivalent fatty acid with the cis/trans ratio preferably being as described hereinafter, n is a number from 1 to three on the weight average in any mixture of compounds, each R<sup>1</sup> in a compound is a C<sub>1-3</sub> alkyl or hydroxy alkyl group, the total of n and the number of R<sup>1</sup> groups that are hydroxyethyl

groups equaling 3, n+m equaling 4, and X is a hair conditioner compatible anion, preferably methyl sulfate. Preferably the cis:trans isomer ratio of the fatty acid (of the C18:1 component) is at least about 1:1, preferably about 2:1, more preferably 3:1, and even more preferably about 4:1, or higher.

The compound, or mixtures of compounds, have (a) either a Hunter "L" transmission of at least about 85, typically from about 85 to about 95, preferably from about 90 to about 95, more preferably above about 95, if possible, (b) only low, relatively non-detectable levels, at the conditions of use, of odorous compounds selected from the group consisting of: isopropyl acetate; 2,2'-ethylidenebis(oxy)bispropane; 1,3,5-trioxane; and/or short chain fatty acid (4-12, especially 6-10, carbon atoms) esters, especially methyl esters; or (c) preferably, both.

The Hunter L transmission is measured by (1) mixing the conditioning active with solvent at a level of about 10% of active, to assure clarity, the preferred solvent being ethoxylated (one mole EO) 2,2,4-trimethyl-1,3-pentanediol and (2) measuring the L color value against distilled water with a Hunter ColorQUEST® colorimeter made by Hunter Associates Laboratory, Reston, Virginia.

The level of odorant is defined by measuring the level of odorant in a headspace over a sample of the conditioning active (about 92% active). Chromatograms are generated using 200 mL of head space sample over about 2 grams of sample. The head space sample is trapped on to a solid absorbent and thermally desorbed onto a column directly via cryofocussing at about -100°C. The identifications of materials is based on the peaks in the chromatograms. Some impurities identified are related to the solvent used in the quaternization process, (e.g., ethanol and isopropanol). The ethoxy and methoxy ethers are typically sweet in odor. There are C<sub>6</sub>-C<sub>8</sub> methyl esters found in the current commercial samples, but not in the typical conditioner actives of this invention. These esters contribute to the perceived poorer odor of the current commercial samples. The level of each odorant found in a typical commercial sample is as follows:

**Approximate concentration of head space impurities**

<u>Chemical Identification</u>	<u>Gas phase concentration (ng/L)</u>	
	<u>Commercial sample</u>	<u>Typical invention sample</u>
Isopropyl acetate	6	< 1
1,3,5-trioxane	61	5
2,2'-ethylidenebis(oxy)-bispropane	244	< 1
C6 methyl ester	10	< 1
C8 Methyl ester	9	< 1
C10 Methyl ester	4	< 1

The acceptable level of each odorant is as follows: isopropyl acetate should be less than about 5, preferably less than about 3, and more preferably less than about 2, nanograms per liter (ng/L.); 2,2'-ethylidenebis(oxy)bispropane should be less than about 200, preferably less than about 100, more preferably less than about 10, and even more preferably less than about 5, nanograms per liter (ng/L.); 1,3,5-trioxane should be less than about 50, preferably less than about 20, more preferably less than about 10, and even more preferably less than about 7, nanograms per liter (ng/L.); and/or each short chain fatty acid (4-12, especially 6-10, carbon atoms) ester, especially methyl esters should be less than about 4, preferably less than about 3, and more preferably less than about 2, nanograms per liter (ng/L.).

The elimination of color and odor materials can either be accomplished after formation of the compound, or, preferably, by selection of the reactants and the reaction conditions. Preferably, the reactants are selected to have good odor and color. For example, it is possible to obtain fatty acids, or their esters, for sources of the long fatty acyl group, that have good color and odor and which have extremely low levels of short chain (C<sub>4-12</sub>, especially C<sub>6-10</sub>) fatty acyl groups. Also, the reactants can be cleaned up prior to use. For example, the fatty acid reactant can be double or triple distilled to remove color and odor causing bodies and remove short chain fatty acids. Additionally, the color of the triethanolamine reactant needs to be controlled to a low color level (e.g. a color reading of about 20 or less on the APHA scale). The degree of clean up required is dependent on the level of use and the presence of other ingredients. For example, adding a dye can cover up some colors. However, for clear and/or light colored products, the color must be almost non-detectable. This is especially true for higher levels of active, e.g., from about 8% to about 75%, preferably from about 13% to about 60%, more preferably from about 18% to about 40%, of the conditioner active by weight of the composition. Similarly, the odor can be covered up by higher levels of perfume, but at the higher levels of conditioner active there is a relatively high cost associated with such an approach, especially in terms of having to compromise the odor quality. Odor quality can be further improved by use of ethanol as the quaternization reaction solvent.

Preferred biodegradable hair conditioning compounds comprise quaternary ammonium salt, the quaternized ammonium salt being a quaternized product of condensation between:

a)-a fraction of saturated or unsaturated, linear or branched fatty acids, or of derivatives of said acids, said fatty acids or derivatives each possessing a hydrocarbon chain in which the number of atoms is between 5 and 21, and

b)-triethanolamine,

characterized in that said condensation product has an acid value, measured by titration of the condensation product with a standard KOH solution against a phenolphthalein indicator, of less than about 6.5.

The acid value is preferably less than or equal to about 5, more preferably less than about 3. Indeed, the lower the AV, the better softness performance is obtained.

The acid value is determined by titration of the condensation product with a standard KOH solution against a phenolphthalein indicator according to ISO#53402. The AV is expressed as mg KOH/g of the condensation product.

For optimum conditioning benefit, it is preferred that the reactants are present in a molar ratio of fatty acid fraction to triethanolamine of from about 1:1 to about 2.5:1.

Preferred cationic, preferably biodegradable quaternary, ammonium hair conditioning compounds can contain the group -(O)CR which is derived from animal fats, unsaturated, and polyunsaturated, fatty acids, e.g., oleic acid, and/or partially hydrogenated fatty acids, derived from vegetable oils and/or partially hydrogenated vegetable oils, such as, canola oil, safflower oil, peanut oil, sunflower oil, corn oil, soybean oil, tall oil, rice bran oil, etc. Non-limiting examples of fatty acids (FA) have the following approximate distributions:

#### Fatty Acyl Group

	FA <sup>1</sup>	FA <sup>2</sup>	FA <sup>3</sup>	FA <sup>4</sup>	FA <sup>5</sup>
C12	trace	trace	0	0	0
C14	3	3	0	0	0
C16	4	4	5	5	5
C18	0	0	5	6	6
C14:1	3	3	0	0	0
C16:1	11	7	0	0	3
C18:1	74	73	71	68	67
C18:2	4	8	8	11	11
C18:3	0	1	1	2	2
C20:1	0	0	2	2	2
C20 and up	0	0	2	0	0
Unknowns	0	0	6	6	7
<i>Total</i>	99	99	100	100	102
IV	86-90	88-95	99	100	95
cis/trans (C18:1)	20-30	20-30	4	5	5
TPU	4	9	10	13	13

TPU is the percentage of polyunsaturates present.



Mixtures of fatty acids, and mixtures of FAs that are derived from different fatty acids can be used, and are preferred. Nonlimiting examples of FA's that can be blended, to form FA's of this invention are as follows:

<u>Fatty Acyl Group</u>	<u>FA<sup>6</sup></u>	<u>FA<sup>7</sup></u>
C14	0	1
C16	11	25
C18	4	20
C14:1	0	0
C16:1	1	0
C18:1	27	45
C18:2	50	6
C18:3	7	0
Unknowns	0	3
<i>Total</i>	100	100
IV	125-138	56
cis/trans (C18:1)	Not Available	7
TPU	57	6

FA<sup>6</sup> is prepared from a soy bean fatty acid, and FA<sup>7</sup> is prepared from a slightly hydrogenated tallow fatty acid.

The more preferred essential hair conditioning actives containing an effective amount of molecules containing two ester linked hydrophobic groups [RC(CO)O-], said actives being referred to hereinafter as "DEQA's", are those that are prepared as a single DEQA from blends of all the different fatty acids that are represented (total fatty acid blend), rather than from blends of mixtures of separate finished DEQA's that are prepared from different portions of the total fatty acid blend.

It is preferred that at least a majority of the fatty acyl groups are unsaturated, e.g., from about 50% to 100%, preferably from about 55% to about 95%, more preferably from about 60% to about 90%, and that the total level of active containing polyunsaturated fatty acyl groups (TPU) be preferably from about 3% to about 30%. The cis/trans ratio for the unsaturated fatty acyl groups is usually important, with the cis/trans ratio being from about 1:1 to about 50:1, the minimum being about 1:1, preferably at least 3:1, and more preferably from about 4:1 to about 20:1. (As used herein, the "percent of conditioner active" containing a given R group is the same as the percentage of that same R group is to the total R groups used to form all of the conditioner actives.)

The unsaturated, including the preferred polyunsaturated, fatty acyl and/or alkylene groups, discussed hereinbefore and hereinafter, surprisingly provide effective conditioning, but also provide better rewetting characteristics, good antistatic characteristics, and especially, superior recovery after freezing and thawing.

The highly unsaturated materials are also easier to formulate into concentrated premixes that maintain their low viscosity and are therefore easier to process, e.g., pump, mixing, etc. These highly unsaturated materials (total level of active containing polyunsaturated fatty acyl groups (TPU) being typically from about 3% to about 30%, with only the low amount of solvent that normally is associated with such materials, i.e., from about 5% to about 20%, preferably from about 8% to about 25%, more preferably from about 10% to about 20%, weight of the total conditioner/solvent mixture, are also easier to formulate into concentrated, stable compositions of the present invention, even at ambient temperatures. This ability to process the actives at low temperatures is especially important for the polyunsaturated groups, since it minimizes degradation. Additional protection against degradation can be provided when the compounds and conditioning compositions contain effective antioxidants, chelants, and/or reducing agents, as disclosed hereinafter.

It will be understood that substituents R and R<sup>1</sup> can optionally be substituted with various groups such as alkoxyl or hydroxyl groups, and can be straight, or branched so long as the R groups maintain their basically hydrophobic character.

A preferred long chain DEQA is the DEQA prepared from sources containing high levels of polyunsaturation, i.e., N,N-di(acyl-oxyethyl)-N,N-methylhydroxyethylammonium methyl sulfate, where the acyl is derived from fatty acids containing sufficient polyunsaturation, e.g., mixtures of tallow fatty acids and soybean fatty acids. Another preferred long chain DEQA is the dioleoyl (nominally) DEQA, i.e., DEQA in which N,N-di(oleoyl-oxyethyl)-N,N-methylhydroxyethylammonium methyl sulfate is the major ingredient. Preferred sources of fatty acids for such DEQAs are vegetable oils, and/or partially hydrogenated vegetable oils, with high contents of unsaturated, e.g., oleoyl groups.

As used herein, when the DEQA diester (n=2) is specified, it can include the monoester (n=1) and/or triester (n=3) that are present. Preferably, at least about 30% of the DEQA is in the diester form, and from 0% to about 30% can be DEQA monoester, e.g., there are three R<sup>1</sup> group. The overall ratios of diester quat to monoester quat are from about 2.5:1 to about 1:1, preferably from about 2.3:1 to about 1.3:1. The level of monoester present can be controlled in manufacturing the DEQA by varying the ratio of

fatty acid, or fatty acyl source, to triethanolamine. The overall ratios of diester quat to triester quat are from about 10:1 to about 1.5:1, preferably from about 5:1 to about 2.8:1.

The above compounds, used as the essential biodegradable quaternized ester-amine conditioning material in the practice of this invention, can be prepared using standard reaction chemistry. In one synthesis of a di-ester variation of DTDMAC, an amine of the formula  $N(CH_2CH_2OH)_3$  is esterified, preferably at two hydroxyl groups, with an acid chloride of the formula  $RC(O)Cl$ , to form an amine which can be made cationic by acidification (one R is H) to be one type of conditioner, or then quaternized with an alkyl halide,  $R^1X$ , to yield the desired reaction product (wherein R and  $R^1$  are as defined hereinbefore). However, it will be appreciated by those skilled in the chemical arts that this reaction sequence allows a broad selection of agents to be prepared.

In preferred DEQA conditioner actives, each R is a hydrocarbyl, or substituted hydrocarbyl, group, preferably, alkyl, monounsaturated alkenyl, and polyunsaturated alkenyl groups, with the conditioner active containing polyunsaturated alkenyl groups being preferably at least about 3%, more preferably at least about 5%, more preferably at least about 10%, and even more preferably at least about 15%, by weight of the total conditioner active present: the actives preferably containing mixtures of R groups, especially within the individual molecules.

In preferred quaternary ammonium compounds, and especially in the DEQAs,  $RC(O)O$  is derived from unsaturated fatty acid, e.g., oleic acid, and/or fatty acids and/or partially hydrogenated fatty acids, derived from animal fats, vegetable oils and/or partially hydrogenated vegetable oils, such as: canola oil; safflower oil; peanut oil; sunflower oil; soybean oil; corn oil; tall oil; rice bran oil; etc.] [As used herein, similar biodegradable actives containing ester linkages are referred to as "DEQA", which includes both diester, triester, and monoester compounds containing from one to three, preferably two, long chain hydrophobic groups. These actives have the characteristic that they can be processed by conventional mixing means at ambient temperature, at least in the presence of about 15% of solvent C, as disclosed hereinbefore.

The DEQAs herein can also contain a low level of fatty acid, which can be from unreacted starting material used to form the DEQA and/or as a by-product of any partial degradation (hydrolysis) of the conditioner active in the finished composition. It is preferred that the level of free fatty acid be low, preferably below about 15%, more preferably below about 10%, and even more preferably below about 5%, by weight of the conditioner active.

The above compounds, used as the quaternized ester-amine conditioning active in the practice of this invention, can be prepared using standard reaction chemistry. In one synthesis of a di-ester variation of DTDMAC, an amine of the formula  $RN(CH_2CH_2OH)_2$  is esterified at both hydroxyl groups with an acid chloride of the formula  $R^1C(O)Cl$ , then quaternized with an alkyl halide,  $RX$ , to yield the desired reaction product (wherein  $R$  and  $R^1$  are as defined hereinbefore). However, it will be appreciated by those skilled in the chemical arts that this reaction sequence allows a broad selection of agents to be prepared.

The actives of the present invention are preferably prepared by a process wherein a chelant, preferably a diethylenetriaminepentaacetate (DTPA) and/or an ethylene diamine-N,N'-disuccinate (EDDS) is added to the process. Also, preferably, antioxidants are added to the fatty acid immediately after distillation and/or fractionation and/or during the esterification reactions and/or prior to, or during, the quaternization reaction, and/or post-added to the finished conditioner active. The resulting conditioner active has reduced discoloration and malodor associated therewith. The typical process comprises the steps of:

a) providing a source of triglyceride and reacting the source of triglyceride to form a mixture of fatty acids and/or fatty acid esters:

b) using the mixture formed from step (a) to react under esterification conditions with triethanolamine;

c) quaternizing, if desired, the mixture of fatty acid esters formed from step (b) by reacting the mixture under quaternizing conditions with a quaternizing agent of the formula  $R^1X$  wherein  $R^1$  is defined as in step (b) and  $X$  is a conditioner compatible anion, preferably selected from the group consisting of chloride, bromide, methylsulfate, ethylsulfate, sulfate, and nitrate thereby forming a quaternary hair conditioning active, the methyl sulfate and ethyl sulfate being highly preferred, wherein at least step (c) is carried out in the presence of a chelating agent selected from the group consisting of diethylenetriaminepentaacetic acid, ethylenediamine-N,N'-disuccinic acid and mixtures thereof.

The step of reacting the source of triglyceride can further include reacting in the presence of the chelating agent step (b) can further include the presence of the chelating agent.

The total amount of added chelating agent is preferably within the range of from about 10 ppm to about 5,000 ppm, more preferably within the range of from about 100 ppm to about 2500 ppm by weight of the formed active. The source of triglyceride is

preferably selected from the group consisting of animal fats, vegetable oils, partially hydrogenated vegetable oils, and mixtures thereof. More preferably, the vegetable oil or partially hydrogenated vegetable oil is selected from the group consisting of canola oil, partially hydrogenated canola oil, safflower oil, partially hydrogenated safflower oil, peanut oil, partially hydrogenated peanut oil, sunflower oil, partially hydrogenated sunflower oil, corn oil, partially hydrogenated corn oil, soybean oil, partially hydrogenated soybean oil, tall oil, partially hydrogenated tall oil, rice bran oil, partially hydrogenated rice bran oil, and mixtures thereof. Most preferably, the source of triglyceride is canola oil, partially hydrogenated canola oil, and mixtures thereof. The process can also include the step of adding from about 0.01% to about 2% by weight of the composition of an antioxidant compound to any or all of steps (a), (b) or (c).

The above processes produce a hair conditioner active with reduced coloration and malodor.

The DEQA actives described hereinabove can contain a low level of the fatty acids which can be unreacted starting material and/or by-product of any partial degradation, e.g., hydrolysis, of the actives in the finished compositions. It is preferred that the level of free fatty acid be low, preferably below about 10%, more preferably below about 5%, by weight of the active.

Particularly preferred conditioning actives and their methods of preparation are disclosed in US Application No. 60/044719.

A commercially available diester quaternary ammonium compound for use herein has the tradename Tetranyl Co-40 and is supplied by Kao. The INCI name for this material is Dioleylethyl Hydroxyethylmonium methosulfate.

A particularly preferred conditioning active for use herein is SC3 which is manufactured according to the synthesis example below.

The quaternary ammonium conditioning agent, is preferably present at a level of from about 1% to about 25%, preferably from about 5% to about 20%, more preferably from about 5% to about 15%, by weight of the conditioning composition.

Detailed Synthesis of new conditioning active "SC3"

Step 1, fatty alcohol compound synthesis: A mixture of about 1,200 grams of the hydrogenated oil from Synthesis Example F (see below) and about 200 grams of the hydrogenated oil from Synthesis Example A (see below) is hydrolyzed three times with about 250°C steam at about 600 psig for about 2.5 hours at a ratio of steam:oil of about 1.2 (by weight). An aqueous solution containing the glycerine which had split off is removed.

The resulting mixture of fatty acids is vacuum distilled for a total of about 150 minutes, in which the pot temperature rose gradually from about 200°C to about 238°C and the head temperature rose gradually from about 175°C to about 197°C. Vacuum of about 0.3-0.6 mm is maintained.

The fatty acids product of the vacuum distillation has an Iodine Value of about 99.1, an amine value (AV) of about 197.6 and a saponification value (SAP) of about 198.6.

step 2)-Esterification:

About 571 grams of Canola fatty acid with an IV of about 100 and an Acid Value of about 196 as made according to the above Fatty Acid Compound Synthesis is added into the reactor, the reactor is flushed with N<sub>2</sub> and about 149 grams of triethanolamine is added under agitation. The molar ratio of fatty acid to triethanol amine is of about 1.875:1. The mixture is heated above about 150° C and the pressure is reduced to remove the water of condensation. The reaction is prolonged until an Acid Value of about 3 is reached.

Step 3)-Quaternization:

To the about 698 grammes of the product of condensation, about 122 grams of dimethylsulfate is added under continuous agitation. The reaction mixture is kept above about 50° C and the reaction is followed by verifying the residual amine value. About 820 grams of conditioner compound of the invention is obtained.

The quaternized material is optionally diluted with e.g. about 15% of an approximately 50:50 ethanol/ hexyleneglycol, preferably more hexylene glycol than ethanol, mixture which lowers the melting point of the material thereby providing a better ease in the handling of the material.

### Other Fatty Acid and Higher Carbon Synthesis Examples

#### Fatty Acid Compound Synthesis Example A

About 1,300 grams of food grade (refined, bleached, degummed) canola oil and approximately 6.5 grams of a commercial nickel hydrogenation catalyst (Engelhard, "N-545"®) corresponding to approximately 0.13 wt.% Ni, are placed in a hydrogenation reactor which is equipped with stirrer. The reactor is sealed and evacuated. The contents are heated to about 170°C and hydrogen is fed into the reactor. Stirring at about 450 rpm is maintained throughout the reaction. After about 10 minutes the temperature in the reactor is about 191°C and the hydrogen pressure is about 11 psig. The temperature is held at about 190°C. After about 127 minutes from when the hydrogen feed began, the hydrogen pressure is about 10 psig. A sample of the reaction mass is drawn and found to have an Iodine Value of about 78 and a cis:trans ratio of about 1.098. After another about 20 minutes at about 190°C, the hydrogen pressure is about 9.8 psig. The hydrogen feed is discontinued and the reactor contents cooled with stirring. The final reaction product has an Iodine Value of about 74.5 and a cis:trans ratio of about 1.35.

The product that forms in the reactor is removed and filtered. It has a cloud point of about 22.2°C. The chain length distributions of the acyl substituents on the sample taken at about 127 minutes, and of the final product, are determined to be as shown in Table 1 in which "sat." means saturated, and "mono" and "di" means monounsaturated and diunsaturated, respectively.

TABLE 1

Chain length	<u>Approximate Percent (mol.)</u>	
	Sample @ 127 min.	Product
C14-sat.	0.1	0.1
C16-sat.	4.7	4.6
C16-mono.	0.4	0.4
C18-sat.	8.9	13.25
C18-mono.	77.0	73.8
C18-di.	4.5	3.1
C20-sat.	0.7	0.75
C-20-mono.	2.1	2.0
Other	1.6	2.0

#### Fatty Acid Compound Synthesis Example B

About 1,300 grams of food grade canola oil and about 5.2 grams of Engelhard "N-545"® nickel hydrogenation catalyst are placed in a hydrogenation reactor which is equipped with a stirrer. The reactor is sealed and evacuated. The contents are heated to about 175°C and hydrogen is fed into the reactor. Stirring is maintained at about 450 rpm throughout the course of reaction. After about 5 minutes the temperature in the reactor is about 190°C and the hydrogen pressure is about 7 psig. The temperature is held at about 190°C. After about 125 minutes from the start of the hydrogen feed, the hydrogen pressure is about 7 psig. A sample of the reaction mass is drawn and found to have an Iodine Value of about 85.4. After another about 20 minutes at about 190°C, the hydrogen pressure is about 6 psig. The hydrogen feed is discontinued and the reactor contents cooled with stirring. The final reaction product has an Iodine Value of about 80. The product that forms in the reactor is removed and filtered. It has a cloud point of about 18.6°C.

#### Fatty Acid Compound Synthesis Example C

About 1,300 grams of food grade canola oil and about 2.9 grams of Engelhard "N-545"® nickel hydrogenation catalyst are placed in a hydrogenation reactor which is equipped with a stirrer. The reactor is sealed and evacuated. The contents are heated to about 180°C and hydrogen is fed into the reactor. Stirring is maintained at about 450 rpm throughout the course of the reaction. After about 5 minutes the temperature in the reactor is about 192°C and the hydrogen pressure is about 10 psig. The temperature is held at about 190 ± 3°C. After about 105 minutes from the start of the hydrogen feed, the hydrogen pressure is about 10 psig. A sample of the reaction mass is drawn and found to have an Iodine Value of about 85.5. After another about 20 minutes at about 190°C, the hydrogen pressure is about 10 psig. The hydrogen feed is discontinued and the reactor contents cooled with stirring. The final reaction product has an Iodine Value of about 82.4. The product that forms in the reactor is removed and filtered. It has a cloud point of about 17.2°C.

#### Fatty Acid Compound Synthesis Example D

About 1,300 grams of food grade canola oil and about 1.4 grams of Engelhard "N-545"® nickel hydrogenation catalyst are placed in a hydrogenation reactor which is equipped with a stirrer. The reactor is sealed and evacuated. The contents are heated to about 180°C and hydrogen is fed into the reactor. After about 5 minutes the temperature in the reactor is about 191°C and the hydrogen pressure is about 10 psig. The



temperature is held at about  $190 \pm 3^\circ\text{C}$ . After about 100 minutes from the start of the hydrogen feed, the hydrogen pressure is about 10 psig. A sample of the reaction mass is drawn and found to have an Iodine Value of about 95.4. After another about 20 minutes at about  $190^\circ\text{C}$ , the hydrogen pressure is about 10 psig. The hydrogen feed is discontinued and the reactor contents cooled with stirring. The final reaction product had an Iodine Value of about 2.3. The product that forms in the reactor is removed and filtered. It has a cloud point of about  $34^\circ\text{C}$ .

#### Fatty Acid Compound Synthesis Example E

About 1,300 grams of food grade canola oil and about 1.3 grams of Engelhard "N-545"® nickel hydrogenation catalyst are placed in a hydrogenation reactor which is equipped with a stirrer. The reactor is sealed and evacuated. The contents are heated to about  $190^\circ\text{C}$  and hydrogen is fed into the reactor to a hydrogen pressure of about 5 psig. After about 3 hours from the start of the hydrogen feed, a sample of the reaction mass is drawn and found to have an iodine value of about 98. The hydrogenation is interrupted, another about 0.7 grams of the same catalyst is added, and the reaction conditions are reestablished at about  $190^\circ\text{C}$  for another about 1 hour. The hydrogen feed is then discontinued and the reactor contents cooled with stirring. The final reaction product had an Iodine Value of about 89.9. The product that forms in the reactor is removed and filtered. It has a cloud point of about  $16^\circ\text{C}$ .

#### Fatty Acid Compound Synthesis Example F

About 1,300 grams of food grade canola oil and about 2.0 grams of Engelhard "N-545"® nickel hydrogenation catalyst are placed in a hydrogenation reactor which is equipped with a stirrer. The reactor is sealed and evacuated. The contents are heated to about  $190^\circ\text{C}$  and hydrogen is fed into the reactor to a hydrogen pressure of about 5 psig. Stirring is maintained at about 420 rpm throughout the course of reaction of the hydrogen feed. After about 130 minutes from the start of the hydrogen feed, the hydrogen feed is discontinued and the reactor contents cooled with stirring. The final reaction product had an Iodine Value of about 96.4. The product that forms in the reactor is removed and filtered. It has a cloud point of about  $11.2^\circ\text{C}$ .

#### Synthesis Example of conditioning compound 1 (SC1)

##### 1)-Esterification:

About 489 grams of partly hydrogenated tallow fatty acid with an IV of about 45 and an Acid Value of about 206, commercially available under the tradename Distal 51 and sold by Witco Corporation, is added into the reactor, the reactor is flushed with N<sub>2</sub> and about 149 grams of triethanolamine is added under agitation. The molar ratio of fatty acid to triethanol amine is of about 1.8:1. The mixture is heated above about 150° C and the pressure is reduced to remove the water of condensation. The reaction is prolonged until an Acid Value of about 5 is reached.

#### 2)-Quaternization:

To about 627 grams of the product of condensation, about 122 grams of dimethylsulfate is added under continuous agitation. The reaction mixture is kept above about 50° C and the reaction is followed by verifying the residual amine value. 749 grams of conditioner compound of the invention is obtained.

The quaternized material is optionally diluted with e.g. about 15% of ethanol which lowers the melting point of the material thereby providing a better handling of the material.

#### Synthesis Example of conditioning compound 2 (SC2)

##### 1)-Esterification:

About 504 grams of oleic fatty acid with an IV of about 90 and an Acid Value of about 198, commercially available under the tradename Emersol 233 and sold by Henkel Corporation, is added into the reactor, the reactor is flushed with N<sub>2</sub> and about 149 grams of triethanolamine is added under agitation. The molar ratio of fatty acid to triethanol amine is about 1.8:1. The mixture is heated above about 150° C and the pressure is reduced to remove the water of condensation. The reaction is prolonged until an Acid Value of about 2 is reached.

##### 2)-Quaternization:

To the about 629 grams of the product of condensation, about 122 grams of dimethylsulfate is added under continuous agitation. The reaction mixture is kept above about 50° C and the reaction is followed by verifying the residual amine value. About 751 grams of conditioner compound of the invention is obtained.

The quaternized material is optionally diluted with e.g. about 8% of ethanol which lower the melting point of the material thereby providing a better ease in the handling of the material.

#### Synthesis Example of Hair Conditioner compound 4 (SC4)

##### 1)-Esterification:

About 457 grams of Canola fatty acid with an IV of about 100 and an Acid Value of about 196, as made according to Fatty Acid Compound Synthesis Example G. is added into the reactor, the reactor is flushed with N<sub>2</sub> and about 149 grams of triethanolamine is added under agitation. The molar ratio of fatty acid to triethanol amine is about 1.6:1. The mixture is heated above about 150° C and the pressure is reduced to remove the water of condensation. The reaction is prolonged until an Acid Value of about 1 is reached.

##### 2)-Quaternization:

To the about 582 grams of the product of condensation, about 122 grams of dimethylsulfate is added under continuous agitation. The reaction mixture is kept above about 50° C and the reaction is followed by verifying the residual amine value. 704 grams of conditioner compound of the invention is obtained.

The quaternized material is optionally diluted with e.g. about 8% of ethanol which lower the melting point of the material thereby providing a better ease in the handling of the material.

The above synthesized compounds have a Hunter L transmission of about 90 and the following levels of odorants in ng/L: Isopropyl acetate < about 1, typically non-detectable; 1,3,5-trioxane about 5.3; 2,2'-ethylidenebis(oxy)-bispropane < about 1, typically non-detectable; C6 methyl ester < about 1, typically non-detectable; C8 Methyl ester < about 1, typically non-detectable; and C10 Methyl ester < about 1, typically non-detectable.

The above synthesized conditioner compound are also exemplified below in the non-limiting hair conditioning composition examples.

#### Abbreviations used in the Examples

In the compositions, the abbreviated component identification have the following meanings:

SC1 : Hair Conditioner compound as made according to Synthesis Example of conditioner compound 1

SC2	:	Hair Conditioner compound as made according to Synthesis Example of conditioner compound 2
SC3	:	Hair Conditioner compound as made according to Synthesis Example of conditioner compound 3
SC4	:	Hair Conditioner compound as made according to Synthesis Example of conditioner compound 4
TMPD	:	2,2,4-trimethyl-1,3-pentanediol
CHDM	:	1,4 cyclohexanedimethanol

The quaternary ammonium conditioning agent, is preferably present at a level of from about 1% to about 25%, preferably from about 5% to about 20%, more preferably from about 5% to about 15%, by weight of the conditioning composition.

#### Principal Solvent System

The conditioning compositions of the present invention also comprise a principal solvent system in addition to water. This is particularly important for formulating liquid, clear hair conditioning compositions. When employed, the principal solvent preferably comprises less than about 40%, preferably from about 5% to about 35%, more preferably from about 5% to about 20%, and even more preferably from about 5% to about 15%, by weight of the composition. The principal solvent is selected to minimize solvent odor impact in the composition and to provide a low viscosity to the final composition. For example, isopropyl alcohol is not very effective and has a strong odor. n-Propyl alcohol is more effective, but also has a distinct odor. Several butyl alcohols also have odors but can be used for effective clarity/stability, especially when used as part of a principal solvent system to minimize their odor. The alcohols are also selected for optimum low temperature stability, that is they are able to form compositions that are liquid with acceptable low viscosities and translucent, preferably clear, down to about 40°F (about 4.4°C) and are able to recover after storage down to about 20°F (about 6.7°C).

Suitable solvents for use herein can be selected based upon their octanol/water partition coefficient (P). Octanol/water partition coefficient of a principal solvent is the ratio between its equilibrium concentration in octanol and in water. The partition coefficients of the principal solvent ingredients of this invention are conveniently given in the form of their logarithm to the base 10, logP.

The logP of many ingredients has been reported; for example, the Pomona92 database, available from Daylight Chemical Information Systems, Inc. (Daylight CIS), Irvine, California, contains many, along with citations to the original literature.

However, the logP values are most conveniently calculated by the "CLOGP" program, also available from Daylight CIS. This program also lists experimental logP values when they are available in the Pomona92 database. The "calculated logP" (ClogP) is determined by the fragment approach of Hansch and Leo (cf., A. Leo, in *Comprehensive Medicinal Chemistry*, Vol. 4, C. Hansch, P. G. Sammens, J. B. Taylor and C. A. Ramsden, Eds., p. 295, Pergamon Press, 1990, incorporated herein by reference). The fragment approach is based on the chemical structure of each ingredient, and takes into account the numbers and types of atoms, the atom connectivity, and chemical bonding. These ClogP values, which are the most reliable and widely used estimates for this physicochemical property, are preferably used instead of the experimental logP values in the selection of the principal solvent ingredients which are useful in the present invention. Other methods that can be used to compute ClogP include, e.g., Crippen's fragmentation method as disclosed in *J. Chem. Inf. Comput. Sci.*, 27, 21 (1987); Viswanadhan's fragmentation method as disclosed in *J. Chem. Inf. Comput. Sci.*, 29, 163 (1989); and Broto's method as disclosed in *Eur. J. Med. Chem. - Chim. Theor.*, 19, 71 (1984). The principal solvents herein are selected from those having a ClogP of from about 0.15 to about 0.64, preferably from about 0.25 to about 0.62, and more preferably from about 0.40 to about 0.60, said principal solvent preferably being at least somewhat asymmetric, and preferably having a melting, or solidification, point that allows it to be liquid at, or near room temperature. Solvents that have a low molecular weight and are biodegradable are also desirable for some purposes. The more asymmetric solvents appear to be very desirable, whereas the highly symmetrical solvents such as 1,7-heptanediol, or 1,4-bis(hydroxymethyl) cyclohexane, which have a center of symmetry, appear to be unable to provide the essential clear compositions when used alone, even though their ClogP values fall in the preferred range.

Operable principal solvents are disclosed and listed below which have ClogP values which fall within the requisite range. These include mono-ols, C6 diols, C7 diols, octanediol isomers, butanediol derivatives, trimethylpentanediol isomers, ethylmethylpentanediol isomers, propyl pentanediol isomers, dimethylhexanediol isomers, ethylhexanediol isomers, methylheptanediol isomers, octanediol isomers, nonanediol isomers, alkyl glyceryl ethers, di(hydroxy alkyl) ethers, and aryl glyceryl ethers, aromatic glyceryl ethers, alicyclic diols and derivatives, C<sub>3</sub>C<sub>7</sub> diol alkoxylated derivatives, aromatic diols, and unsaturated diols. Particularly preferred principal solvents include hexanediols such as 1,2-Hexanediol and 2-Ethyl-1,3-hexanediol and pentanediols such as 2,2,4-Trimethyl-1,3-pentanediol. These principal solvents are all disclosed in copending U.S. Patent application numbers 08/621,019; 08/620,627;

08/620,767; 08/620,513; 08/621,285; 08/621,299; 08/621,298; 08/620,626; 08/620,625; 08/620,772; 08/621,281; 08/620,514; and 08/620,958, all filed March 22, 1996 and all having the title "CONCENTRATED, STABLE, PREFERABLY CLEAR, FABRIC SOFTENING COMPOSITION", and WO98/47991, the disclosures of which are all herein incorporated by reference.

Especially preferred for use in the hair conditioning compositions herein is 1,2-hexanediol.

#### 1,3-butylene glycol

The conditioning compositions of the present invention also comprise from about 0.1% to about 10%, preferably from about 1% to about 5%, more preferably from about 2% to about 4%, by weight of the composition of 1,3-butylene glycol in addition to the principal solvent mentioned above. The 1,3-butylene glycol is preferably mixed with the DEQA to help to improve clarity and stability of the composition.

#### Optional Ingredients

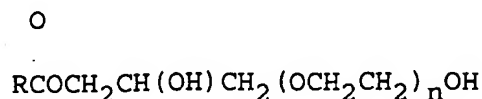
The conditioning compositions herein may include a wide variety of additional ingredients, non-limiting examples of which are given below.

The conditioning compositions herein can comprise an emollient selected from polyethylene glycol derivatives of glyceride, polypropylene and polyethylene glycol ethers of glucose and polypropylene glycol ethers of fatty alcohol, and mixtures thereof, preferably a water-soluble emollient. The compositions preferably comprise from about 0.1% to about 10%, preferably 0.1% to about 5%, by weight, of the emollient.

#### Polyethylene glycol derivatives of glycerides

Suitable polyethylene glycol derivatives of glycerides include any polyethylene glycol derivative of glycerides which are water-soluble and which are suitable for use in a hair conditioning composition. Suitable polyethylene glycol derivatives of glycerides for use herein include derivatives of mono-, di- and tri-glycerides and mixtures thereof.

One class of polyethylene glycol derivatives of glycerides suitable herein are polyethyleneglycol glyceryl fatty esters having the formula (1):



wherein n, the degree of ethoxylation, is from about 4 to about 200, preferably from about 5 to about 100, more preferably from about 6 to about 80, and wherein R comprises an aliphatic radical having from about 5 to about 25 carbon atoms, preferably from about 7 to about 20 carbon atoms.

Suitable polyethylene glycol derivatives of glycerides include PEG-20 almond glycerides, PEG-60 almond glycerides, PEG-11 avocado glycerides, PEG-6 capric/caprylic glycerides, PEG-8 capric/caprylic glycerides, PEG-20 corn glycerides, PEG-60 corn glycerides, PEG-60 evening primrose glycerides, PEG-7 glyceryl cocoate, PEG-30 glyceryl cocoate, PEG-40 glyceryl cocoate, PEG-78 glyceryl cocoate, PEG-80 glyceryl cocoate, PEG-12 glyceryl dioleate, PEG-15 glyceryl isostearate, PEG-20 glyceryl isostearate, PEG-30 glyceryl isostearate, PEG-75 cocoa butter glycerides, PEG-20 hydrogenated palm oil glycerides, PEG-70 mango glycerides, PEG-13 mink glycerides, PEG-75 shorea butter glycerides, PEG-10 olive glycerides, PEG-12 palm kernal glycerides, PEG-45 palm kernal glycerides, PEG-8 glyceryl laurate and PEG-30 glyceryl laurate. Mixtures of polyethylene glycol derivatives of glycerides can also be used herein.

Preferred for use herein is a polyethylene glycol derivative of sunflower oil commercially available from Floratech under the tradename Florasun PEG-10.

#### Polyalkylene glycol ether of a glucose

Suitable polyalkylene glycol ethers of glucose for use herein include any water-soluble polyalkylene glycol ether of glucose suitable for use in a hair conditioning composition. Preferred herein are polyethylene glycol ether and polypropylene glycol ethers of glucose. Suitable examples include PPG-10 methylglucose ether, PPG-20 ethyl glucose ether, Methyl Gluceth-20 and Methyl Gluceth-10. Mixtures of polyalkylene glycol ethers of glucose can also be used herein.

Polypropylene glycol ether of fatty alcohol

Suitable polypropylene glycol ethers of fatty alcohol for use herein include any water-soluble polypropylene glycol ether of fatty alcohol suitable for use in a hair conditioning composition. Suitable examples include PPG-3 myristylether and PPG-2 myristyl ether propionate. Mixtures of polypropylene glycol ethers of fatty alcohols can also be used herein.

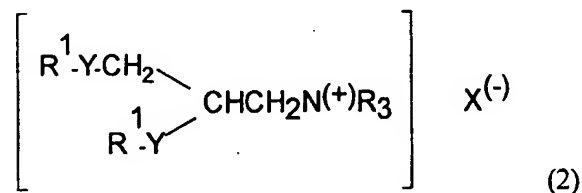
Most preferred for use in the compositions herein is a polypropylene glycol ether of a glyceride.

Additional Conditioning Actives

The conditioning compositions herein may comprise other hair conditioning actives in addition to the DEQA of formula (1) as described above.

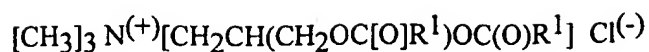
Other suitable conditioning agents for use herein include, but are not limited to, quaternary ammonium conditioning agents, such as ester substituted quaternary ammonium compounds, amide substituted quaternary ammonium compounds and alkyl substituted quaternary ammonium compounds such as those quaternary ammonium compounds disclosed in US-A-5,610,187 (Witco) incorporated herein by reference, mixed amide/ester substituted quaternary ammonium compounds such as those disclosed in EP-A-682935 (Kao) incorporated herein by reference and protonated amines.

A second type of DEQA active has the general formula:



wherein each Y, R, R<sup>1</sup>, and X<sup>(-)</sup> have the same meanings as before. Such compounds include those having the formula:





where each R is a methyl or ethyl group and preferably each R<sup>1</sup> is in the range of C<sub>15</sub> to C<sub>19</sub>. As used herein, when the diester is specified, it can include the monoester that is present. The amount of monoester that can be present is the same as in DEQA (1).

These types of agents and general methods of making them are disclosed in U.S. Pat. No. 4,137,180, Naik et al., issued Jan. 30, 1979, which is incorporated herein by reference. An example of a preferred DEQA of formula (2) is the "propyl" ester quaternary ammonium active having the formula 1,2-di(acyloxy)-3-trimethylammonio propane chloride, where the acyl is the same as that of DEQA<sup>5</sup>, and is denoted hereinafter as DEQA<sup>9</sup>.

#### Other Optional Components

In addition to the principal solvent system and the 1,3-butylene glycol, the compositions herein may also comprise additional water soluble solvents, e.g., ethanol; isopropanol; 1,2-propanediol; 1,3-propanediol and propylene carbonate.

The hair conditioning compositions herein can also comprise a wide variety of additional ingredients which are known for use in conventional hair conditioning compositions, non-limiting examples of which are given below.

The conditioning compositions herein may contain one or more monohydric fatty alcohols. Suitable fatty alcohols for use herein are fatty alcohols having a melting point of 30°C or lower being preferably selected from unsaturated straight chain fatty alcohols, saturated branched chain fatty alcohols, saturated C<sub>8</sub>-C<sub>12</sub> straight chain alcohols, and mixtures thereof. When present the fatty alcohol is preferably used at a level of from about 0.1% to about 10%, by weight, preferably from about 0.1% to about 5%, more preferably from about 0.25% to about 1%.

The unsaturated straight chain fatty alcohols will typically have one degree of unsaturation. Di- and tri- unsaturated alkenyl chains may be present at low levels, preferably less than about 5% by total weight of the unsaturated straight chain fatty alcohol, more preferably less than about 2%, most preferably less than about 1%.

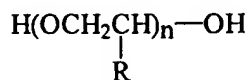
Preferably, the unsaturated straight chain fatty alcohols will have an aliphatic chain size of from C<sub>12</sub>-C<sub>22</sub>, more preferably from C<sub>12</sub>-C<sub>18</sub>, most preferably from C<sub>16</sub>-C<sub>18</sub>. Especially preferred alcohols of this type include oleyl alcohol and palmitoleic alcohol.

The branched chain alcohols will typically have aliphatic chain sizes of from C<sub>12</sub>-C<sub>22</sub>, preferably C<sub>14</sub>-C<sub>20</sub>, more preferably C<sub>16</sub>-C<sub>18</sub>. Exemplary branched chain alcohols for use herein include isostearyl alcohol, octyl dodecanol, and octyl decanol.

Examples of saturated C<sub>8</sub>-C<sub>12</sub> straight chain alcohols include octyl alcohol, caprylic alcohol, decyl alcohol, and lauryl alcohol.

The present compositions are preferably limited to levels of fatty alcohols, such as cetyl alcohol and stearyl alcohol, of no more than about 5%, preferably no more than about 1%, more preferably 0%, by weight of the composition.

The conditioning compositions herein may also comprise from about 0.1% to about 10%, by weight, preferably from about 0.2% to about 5%, more preferably from about 0.5% to about 3%, of a polymer of ethylene oxide, propylene oxide, and mixtures thereof, having the general formula:



wherein R is selected from the group consisting of H, methyl, and mixtures thereof; and n has an average value of from about 2,000 to about 14,000, preferably from about 5,000 to about 9,000, more preferably from about 6,000 to about 8,000. When R is H, these materials are polymers of ethylene oxide, which are also known as polyethylene oxides, polyoxyethylenes, and polyethylene glycols. When R is methyl, these materials are polymers of propylene oxide, which are also known as polypropylene oxides, polyoxypropylenes, and polypropylene glycols. When R is methyl, it is also understood that various positional isomers of the resulting polymers can exist. In the above structure, n has an average value of from about 2,000 to about 14,000, preferably from about 5,000 to about 9,000, more preferably from about 6,000 to about 8,000.

Polyethylene glycol polymers useful herein that are especially preferred are PEG-2M wherein R equals H and n has an average value of about 2,000 (PEG 2-M is also known as Polyox WSR® N-10 from Union Carbide and as PEG-2,000); PEG-5M wherein R

equals H and n has an average value of about 5,000 (PEG 5-M is also known as Polyox WSR® N-35 and Polyox WSR® N-80, both from Union Carbide and as PEG-5,000 and Polyethylene Glycol 300,000); PEG-7M wherein R equals H and n has an average value of about 7,000 (PEG 7-M is also known as Polyox WSR® N-750 from Union Carbide); PEG-9M wherein R equals H and n has an average value of about 9,000 (PEG 9-M is also known as Polyox WSR® N-3333 from Union Carbide); and PEG-14 M wherein R equals H and n has an average value of about 14,000 (PEG 14-M is also known as Polyox WSR® N-3000 from Union Carbide.)

Other useful polymers include the polypropylene glycols and mixed polyethylene/polypropylene glycols.

The compositions herein can comprise conditioning agents in addition to the quaternary ammonium conditioning agents described hereinabove. Suitable conditioning agents include cationic surfactants, cationic polymers, nonvolatile silicones, nonvolatile hydrocarbons, saturated C<sub>14</sub> to C<sub>22</sub> straight chain fatty alcohols, nonvolatile hydrocarbon esters, and mixtures thereof. Suitable conditioning agents are disclosed in WO95/20939 which is incorporated herein by reference.

A wide variety of additional ingredients can be formulated into the present conditioning compositions. These include hair-hold polymers, deterative surfactants such as anionic, nonionic, amphoteric, and zwitterionic surfactants, additional viscosity modifying agents and suspending agents such as xanthan gum, guar gum, hydroxypropyl guar, hydroxyethyl cellulose, methyl cellulose, hydroxyethylcellulose, starch and starch derivatives; insoluble and soluble silicones (eg. dimethicone copolyol); viscosity modifiers such as methanolamides of long chain fatty acids such as cocomonoethanol amide; crystalline suspending agents; pearlescent aids such as ethylene glycol distearate; preservatives such as benzyl alcohol, methyl paraben, propyl paraben and imidazolidinyl urea; polyvinyl alcohol; ethyl alcohol; pH adjusting agents, such as citric acid, sodium citrate, succinic acid, phosphoric acid, sodium hydroxide, sodium carbonate; salts, in general, such as potassium acetate and sodium chloride; coloring agents, such as any of the FD&C or D&C dyes; hair oxidizing (bleaching) agents, such as hydrogen peroxide, perborate and persulfate salts; hair reducing agents, such as the thioglycolates; perfumes; sequestering agents, such as disodium ethylenediamine tetra-acetate; and polymer plasticizing agents, such as glycerin, disobutyl adipate, butyl stearate, and propylene glycol. Such optional ingredients generally are used individually at levels from about

0.01% to about 10.0%, preferably from about 0.05% to about 5.0% by weight of the composition.

The conditioning compositions herein can be formulated in a wide variety of product forms, including but not limited to creams, gels, foams, mousses and sprays. Mousses, foams and sprays can be formulated with propellants such as propane, butane, pentane, dimethylether, hydrofluorocarbon, CO<sub>2</sub>, N<sub>2</sub>O, or without propellants (using air as the propellant in a pump spray or pump foamer package).

#### METHOD OF USE

The conditioning composition herein may be used in a conventional manner for conditioning human hair or skin. Preferably after shampooing, an effective amount of the conditioner composition, typically from about 1 gram to about 50 grams, preferably from about 1 gram to about 20 grams, is then applied to the hair or skin. Application of the conditioner typically includes working the composition through the hair, generally with the hands and fingers, or with a suitable implement such as a comb or brush. The conditioner composition is preferably then rinsed from the hair or skin with water, but can also be left on the hair.

The preferred method for conditioning the hair therefore comprises the steps of:

- (a) applying an effective amount of the conditioning composition to hair which has been shampooed,
- (b) working the conditioning composition into the hair with hands and fingers or with a suitable implement,
- (g) rinsing the conditioning composition from the hair using water.

These steps can be repeated as many times as desired to achieve the conditioning benefit sought, preferably only once.

In an alternative method herein the conditioner herein can be left on the hair instead of being rinsed out.

EXAMPLES

The following examples further describe and demonstrate embodiments within the scope of the present invention.

Preparation

The conditioning compositions of the present invention can be prepared by using conventional mixing and formulating techniques.

Conditioning Compositions I-IV

			Foam	Spray	Mousse	Opacified Gel
Ingredient			I/%	II/%	III/%	IV/%
Tetranyl Co-40 (80% active) <sup>1</sup>			12.5	3.1	12.5	18
1,2-Hexanediol			10	10	10	5
Methylparaben			0.2	0.2	0.2	0.2
Propylparaben			0.1	0.1	0.1	0.1
1,3 Butylene glycol			2	2	2	2
Perfume			1	1	1	1
Dimethicone copolyol			0.1	0.1	0.1	0.1
SF1188A <sup>5</sup>						
Florasun PEG-10 <sup>2</sup>			2	2	2	2
Jaguar HP-105 <sup>3</sup>			0	0	0	1
TiO <sub>2</sub> (40% in 1,3-butylene glycol)			0	0	0	0.25
AP 30 Propellant <sup>4</sup>			0	0	4.3	0
Water			-----to 100-----			
			Foam	Spray	Mousse	Opacified Gel
Ingredient			I/%	II/%	III/%	IV/%
Tetranyl Co-40 (80% active) <sup>1</sup>			12.5	12.5	12.5	18

1,2-Hexanediol	10	2.5	10	5
Methylparaben	0.2	0.2	0.2	0.2
Propylparaben	0.1	0.1	0.1	0.1
1,3 Butylene glycol	2	2	2	2
Perfume	1	1	1	1
Dimethicone copolyol	0.1	0.1	0.1	0.1
SF1188A <sup>5</sup>				
Florasun PEG-10 <sup>2</sup>	2	2	2	2
Jaguar HP-105 <sup>3</sup>	0	0	0	1
TiO <sub>2</sub> (40% in 1,3-butylene glycol)	0	0	0	0.25
AP 30 Propellant <sup>4</sup>	0	0	4.3	0
hexylene glycol	0.75	0.75	0.75	0.75
ethanol	0.75	0.75	0.75	0.75
Water	-----to 100-----			

1. Supplied by Kao
2. Supplied by Floratec
3. Supplied by Rhone Poulenc
4. Mixture of propane, isobutane and n-butane, supplied by BP
5. Supplied by GE Silicones

#### Process for preparation of conditioner liquid/gel

The Tetranyl Co-40 and hexanediol are added to the mixing vessel. Agitation is begun slowly. The butylene glycol is added and heated to 40°C. Agitation is continued and the methyl and propyl paraben is added at 40°C and allowed to dissolve. The composition is then cooled. With agitation the remaining ingredients are added while cooling. The perfume is added at about 25°C. The water is added and mixed until homogeneous. The pH is adjusted to 3.5 with sodium hydroxide.

#### Process for preparation of foam

The resulting liquid is put into a pump foamer package such as AIRSPRAY supplied by Zuiderkade.

Process for preparation of aerosol mousse

To a 150ml aluminium can, 132ml of the above concentrate is added. The can is crimped and a vacuum is drawn. 6g of propellant is added.

Process for preparation of spray

The resulting liquid from the process for preparation of the mousse is put into a suitable spray gel can.

Examples VI-VII (Pump Foamer Hair Conditioning Compositions)

Ingredient	VI/%	VII/%
SC3 <sup>1</sup>	7.5	7.5
1,2-hexanediol	8.0	7.5
1,3-butylene glycol	2	2
hexylene glycol	2.6	2.66
PEG-10 Sunflower glyceride	0.0	5.0
PEG-20 Glyceryl laurate	4.0	0.0
Dimethicone copolyol	1.0	0.0
PEG-2M	0.6	0.4
PEG-5M	0.4	0.6
Perfume	1	1
Preservative	<1	<1
Minors/by products	<1	<1
Water	to 100	to 100

1. SC3 prepared according to Synthesis Example SC3 hereinabove.

The pH target for the compositions of Example VI-VII is pH 3-4 which is adjusted using sodium hydroxide (32%/1.0N). The Examples VI-VII can be prepared and packaged using conventional techniques such as those used hereinabove in the pump foamer Examples.

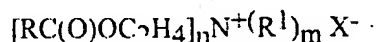
The conditioning compositions of the examples provide excellent hair detangling and wet clean feel on rinsing and post rinsing with the additional benefits of minimizing volume loss.



What is claimed is:

1. A hair conditioning composition comprising:

- (a) a quaternary ammonium cationic conditioning agent having the formula (I):



wherein each R in a compound is a C<sub>6</sub>-C<sub>22</sub>, preferably with only minimal, or no, C<sub>6</sub>-10 hydrocarbyl group, preferably having an Iodine Value (hereinafter also referred to as IV) of from about 70 to about 140 based upon the IV of the equivalent fatty acid, n is a number from 1 to three on the weight average in any mixture of compounds, each R<sup>1</sup> in a compound is a C<sub>1-3</sub> alkyl or hydroxy alkyl group, the total of n and the number of R<sup>1</sup> groups that are hydroxyethyl groups equaling 3, n+m equaling 4, and X is a hair conditioner compatible anion, preferably methyl sulfate, the conditioning agent preferably having (a) a Hunter L transmission of at least about 85, (b) non-detectable levels at the conditions of use of odorous compounds selected from isopropyl acetate, 2,2'-ethylidenebis(oxy)bispropane, and/or short fatty acid esters or (c) preferably both, wherein preferably the level of conditioning agent containing polyunsaturated alkylene groups being at least about 3% by weight of the total conditioning agent present and preferably wherein the conditioning agent comprises a mixture of monoester and diester;

- (b) a mono-ol or diol solvent having a ClogP of from about 0.15 to about 0.64; and

- (c) 1,3-butylene glycol.

2. A hair conditioning composition according to Claim 1 wherein the conditioning composition comprises from about 0.1% to about 20%, preferably from about 0.5% to about 15%, by weight, of quaternary ammonium conditioning agent.
3. A hair conditioning composition according to Claim 1 or 2 comprising from about 0.1% to about 20%, preferably from about 0.5% to about 15% by weight of the mono-ol or diol solvent.
4. A hair conditioning composition according to any of Claims 1 to 3 wherein the mono-ol or diol solvent is 1,2-hexanediol.

5. A hair conditioning composition according to any of Claims 1 to 4 comprising from about 0.1% to about 5% by weight of 1,3-butylene glycol.
6. A composition according to any of Claims 1 to 5 wherein each R substituent has a cis:trans ratio (for the C18:1 component) of from about 1:1 to about 20:1 based upon the equivalent fatty acid.
7. A composition according to any of Claims 1 to 6 wherein the Hunter L transmission is from about 85 to about 95, preferably from about 90 to 95.
8. A method according to any of Claims 1 to 7 additionally comprising a thickening agent selected from hydroxyalkylguar thickening agents, preferably hydroxypropyl guar.

# INTERNATIONAL SEARCH REPORT

International Application No.

PCT/US 99/14715

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A61K7/50

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 96 03970 A (WITCO) 15 February 1996 (1996-02-15) claims 1,6-11 page 6, line 29 -page 7, line 26 page 9 page 10, line 3-23 page 11, line 10-14 page 12, line 4-10	1,2,5
A	EP 0 655 236 A (L'OREAL) 31 May 1995 (1995-05-31) claims 1,2,12,18,30,32 page 4, column 30-35 page 5, column 30-34	1,2,5



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

### \* Special categories of cited documents:

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

20 October 1999

Date of mailing of the international search report

17/11/1999

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# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 99/14715

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication where appropriate, of the relevant passages	Relevant to claim No.
P,X	<p>WO 98 47991 A (PROCTER &amp; GAMBLE)  29 October 1998 (1998-10-29)  claims 1-5  page 19, line 18 -page 21, line 10  page 21, line 34 -page 22, line 4  page 22, line 37 -page 23, line 9  page 25, line 30 -page 26, line 21  page 28, line 4-39  page 67, line 23-38  -----</p>	1-8

# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 99/ 14715

## Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
  
2. ☒ Claims Nos.:  
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:  
  
see FURTHER INFORMATION sheet PCT/ISA/210
  
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
  
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
  
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
  
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims: it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Claims 1 and 7 are dealing with physical parameters. As search on physical parameters is not possible, these parameters have not been taken into consideration during the search.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 99/14715

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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WO 9847991 A	29-10-1998	NONE	